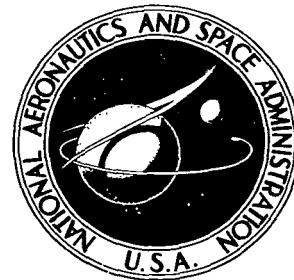


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VOLATILIZATION OF OXIDES DURING OXIDATION OF SOME SUPERALLOYS AT 1200° C

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16. Abstract Volatilization of oxides during cyclic oxidation of commercial Nichrome, Inconel 750, René 41, Stellite 6B, and GE-1541 was studied at 1200° C in static air. Quantitative analysis of oxide vapor deposits revealed that oxides of tungsten, molybdenum, niobium, manganese, and chromium volatilized preferentially from the oxide scales. Aluminum and silicon were not detected in vapor deposits. For all the alloys except GE-1541, chromium was found to be the main metallic element in the oxide scales.	13. Type of Report and Period Covered Technical Note		
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VOLATILIZATION OF OXIDES DURING OXIDATION OF SOME SUPERALLOYS AT 1200° C

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SUMMARY

The subject of this study was the volatilization of oxides during the oxidation of commercial Nichrome, Inconel 750, René 41, Stellite 6B, and GE-1541 at 1200° C in static air. For this purpose a cold-wall furnace technique was used whereby the specimens were heated inductively and the oxide vapors were collected on a cold platinum foil. The composition of the vapor deposit was determined *in situ* by the X-ray fluorescent technique. The results revealed that the oxides of tungsten, molybdenum, niobium, manganese, and chromium were preferentially lost from the oxide scales by volatilization. Aluminum and silicon were not detected in the oxide vapor deposits of alloys containing these elements. The concentrations of nickel, cobalt, and iron - the main component elements of the alloys - were considerably lower in the vapor deposits and in the oxide scales than in the alloys themselves.

INTRODUCTION

One of the factors that can destroy or modify the protective nature of oxide scales is volatilization. In general, volatilization increases significantly with the temperature and the gas flow rate. Thus, its detrimental effects become more important in advanced high-temperature, high-flow, air-breathing propulsion systems. In the numerous studies that have been made of the oxidation of superalloys, the emphasis has been placed primarily on the reaction kinetics of the process and on the identification and morphology of the oxide-scale constituents. The phenomenon of volatilization has been largely ignored. The exceptions are the studies (refs. 1 and 2) concerned with the volatilization of chromium oxide during the oxidation of chromium-containing alloys. However, these studies were somewhat misleading because they suggested that only this oxide volatilized during the oxidation of such alloys. It is reasonable to expect that other metallic oxides

would volatilize also. This has been demonstrated by Kohl and Stearns (ref. 3). They reported that the major products of volatilization during the oxidation of two silicide-coated niobium alloys contained chromium, silicon, iron, and tungsten.

The purpose of this study was to determine experimentally the proportions of metallic elements in oxide vapor deposits obtained during the oxidation of a variety of super-alloys in still air at atmospheric pressure and 1200° C, which is near their upper use-temperature. The experimental technique used in this study was based on inductive heating of a specimen and quantitative analysis of oxides deposited from the vapor on cold platinum foil, which surrounded the specimen.

EXPERIMENTAL PROCEDURE

Sample Preparation

In order to examine a broad range of alloy compositions, the following alloys were used in this study: Inconel 750 (registered trademark of International Nickel Co.), René 41 (registered trademark of General Electric Co.), Stellite 6B (registered trademark of Cabot-Stellite Corp.), GE-1541 (registered trademark of General Electric Co.), and commercial Nichrome. Their compositions are shown in tables I to IV or in the text (GE-1541). They were obtained in the form of 1.27-centimeter-diameter rods and machined into cylindrical specimens 1.27 centimeters high and of the same diameter with a central concentric blackbody hole. The surface of each specimen was finished with 320-grit emery paper, and its edges were given a small radius by grinding. Prior to testing, the specimens were degreased and ultrasonically cleaned.

Apparatus and Oxidation-Volatilization Experiments

Oxidation-volatilization experiments were carried out in a cold-wall furnace, a schematic illustration of which is shown in figure 1.

A specimen supported by a three-pronged alumina rod was contained in a vertical quartz tube. The central portion of the tube, about 5 centimeters long, was wrapped by an induction coil. The inside surface of the tube, just about opposite the induction coil, was lined with properly shaped platinum foil as a collector for the condensing oxide vapors. The distance between the surface of the specimen and the collector was 0.3 centimeter. The quartz tube and platinum collector were kept cool by a water jacket with circulating tap water. The induction coil, which was designed to couple inductively with the specimen, was connected to a 2.5-kilowatt, high-frequency induction heating generator. The specimen was heated to the desired temperature by controlling the power

input. Occasional adjustment of the power input was necessary to maintain constant temperature. Temperature determinations were made with a disappearing-filament micro-optical pyrometer calibrated against a standard tungsten strip lamp. In order to avoid fogging the flat quartz window (and thus affecting temperature determinations), the furnace was provided with a shutter, which was opened only when the temperature measurements were made. Unless otherwise specified the oxidation-volatilization experiments were run at 1200° C for four 8-hour-long cycles. After each 8-hour cycle the platinum collector was removed from the quartz tube for analysis of the deposit and replaced with a new one.

After two specimens of each material had been exposed to four oxidation cycles, the three-pronged alumina support rod was replaced with a clean one and the inner surfaces of the quartz window and of the quartz tube were cleaned. The tests for each alloy were run in duplicate and the analytical data for the vapor deposits, shown in tables I to IV, are averages of two determinations.

Analysis

The content of metallic elements in the oxide deposits that were collected on the platinum foil was studied in situ, without removing them from the foil, by X-ray fluorescent analysis with a high-resolution, solid-state X-ray detector. The signals from the detector were fed directly into a computer and analyzed with the aid of the SAVLOC program (ref. 4). This technique permitted a rapid quantitative analysis of the relatively small amounts of material collected. The technique was satisfactorily verified on two standard samples of known composition. The total amount of the deposit was rather small and varied from alloy to alloy, larger deposits being produced by alloys containing tungsten and molybdenum. The deposits never exceeded a few milligrams in weight. The platinum collectors were reused after deposits were fluxed with sodium borate and washed in boiling distilled water.

After completion of the fourth oxidation cycle, the retained oxide scales were also analyzed in situ for constituent oxides by X-ray diffraction and for elements by X-ray fluorescence on small amounts of scraped scale. After these analyses the specimens were sectioned perpendicularly to their axes, polished, etched, and metallographically examined and photographed. Finally, some of the polished specimens were used for microhardness (DPH) determinations with a 200-gram load to reveal the hardness changes in the superalloys near the surface as a result of preferential loss of some component elements during oxidation.

RESULTS

Commercial Nichrome

Table I summarizes the analytical data obtained for commercial Nichrome, a simple Ni-Cr alloy. The proportions of metals in the retained scale and in the vapor deposits are quite different from the nominal composition of Nichrome. The composition of the vapor deposits changed with time. The nickel content in the alloy was 78.9 wt %, but in the scale it was only 14.3 wt %. It was even lower in the vapor deposit, decreasing with time from 7.0 wt % during the first cycle to 4.8 wt % during the fourth cycle. The chromium content showed an opposite trend. It increased from 19.26 wt % in the alloy to 67.3 wt % in the scale and to 81.4 wt % in the vapor deposit collected during the fourth cycle. The concentration of manganese was 10 times greater in the scale than in the alloy. It reached 29.4 wt % in the vapor deposit after the first oxidation cycle. The phases forming the retained scale were Cr_2O_3 and spinel with a lattice parameter a_0 of 8.45 Å. This spinel must have contained some manganese because the lattice parameter of pure NiCr_2O_4 is 8.32 Å and no oxides of manganese were detected by X-ray. Metallographic examination of a polished specimen that was subjected to four oxidation cycles (fig. 2) revealed the presence of porosity near the surface. The originally small alloy grains underwent a significant growth, as would be expected from such a high-temperature exposure. Microhardness tests did not reveal any softening in the depletion zone.

Inconel 750

Inconel 750, containing nine constituent elements (table II) exhibited quite different chromium volatilization from the oxide scale than was observed for Nichrome. Chromium content in the vapor deposit was 86.2 wt % during the first oxidation cycle and declined gradually to 64.3 wt % during the fourth cycle. The chromium content increased from 15.0 wt % in the alloy to 46.2 wt % in the scale. The vapor deposit collected during the first oxidation showed only 4.4-wt % Ni, but this gradually increased to 24 wt % during the fourth cycle. Iron was present in the scale and in the vapor deposits, in amounts approximately similar to those in the alloy. Titanium was found in the oxide scale but not in the vapor deposits. Conversely, niobium was not detected in the scale but appeared in the vapor deposits. During the first three cycles, the manganese content was higher in the scale and in the vapor deposits than in the alloy. Apparently, the contents of aluminum and silicon in the scale were too low to be detected. Neither of the two elements was detected in the vapor deposits.

It is obvious that carbon could not be detected in the scale or in vapor deposits because it oxidized to form a gas. The phases that were identified in the retained oxide scale were Cr_2O_3 , spinel ($a_0 = 8.40 \text{ \AA}$), and rutile. A metallographically prepared cross section of an oxidized specimen (fig. 3) revealed the existence, near the surface, of a 0.05-centimeter-thick depletion zone and the presence of porosity, which went considerably deeper. Again, very significant grain growth took place during the four oxidation cycles at 1200°C . Microhardness determinations indicated a very pronounced but gradual softening of the alloy in a zone that was 0.1 centimeter deep (fig. 4).

René 41

Due to "explosive" spallation of the oxide scale on René 41 during the cooling part of the oxidation cycles, it was possible that vapor deposits from this alloy would be contaminated by minute fragments of the scale. Therefore, it was necessary to remove the platinum collector from the quartz tube immediately after the heating period of the oxidation cycle was completed and while the specimen was still red hot. The results of the analysis of these vapor deposits are shown in table III. Chromium and nickel were the predominant elements in the oxide scale. The chromium content was 67.9 wt % in the vapor deposit collected during the first oxidation cycle, and it steadily decreased to 5.7 wt % during the fourth cycle. The amount of nickel in the vapor deposits was considerably lower than in the alloy and it never exceeded 2 wt %. Although René 41 contained 11-wt % cobalt, only traces of cobalt were detected during the first and third cycles. None of it was detected in deposits collected during the second and fourth cycles.

Molybdenum was the predominant component in deposits collected during the second, third, and fourth oxidation cycles. The molybdenum content increased to 94.3 wt %, which was 9.43 times greater than its content in the alloy. The concentration of titanium in the deposit collected during the first cycle was higher than that in the alloy, but it decreased rapidly with time and titanium was not detected during the fourth cycle. Aluminum was not found in the retained scale or in the deposits. An X-ray diffraction analysis of the retained scale indicated the presence of Cr_2O_3 , spinel ($a_0 = 8.30 \text{ \AA}$), and rutile structure. Examination of a metallographically prepared cross section of an oxidized specimen (fig. 5) revealed the presence of porosity to a depth of 0.05 centimeter and considerable grain growth. A very significant drop in microhardness was noted in a zone approximately 0.04 centimeter deep (fig. 4).

Stellite 6B

As in the cases of previously discussed alloys, the concentration of chromium was greater in the retained oxide scale on Stellite 6B than in the alloy itself (table IV). It was also greater in the vapor deposit collected during the first oxidation cycle. However, in the subsequent deposits, the chromium content decreased, being 20.8 wt % in the fourth deposit. The amount of cobalt in the retained scale and in the vapor deposits was considerably lower than that in the alloy, and it decreased with time. Iron was not detected in the vapor deposits. The concentrations of tungsten and molybdenum were considerably greater in the vapor deposits than in the alloy or retained scale, and they increased with time. The apparent anomaly in the concentration values of the component elements, mainly tungsten and molybdenum, obtained during the fourth oxidation cycle was due to oxide-scale spallation during the cooling period of the third cycle.

Metallographic examination of this alloy after oxidation (fig. 6) revealed a moderate growth of the matrix grains and a decrease in the quantity of carbide precipitates (M_7C_3 and $M_{23}C_6$, where M represents the sum of carbide-forming elements). This depletion zone was about 0.1 centimeter deep. Just below the surface a small amount of porosity could be observed. An X-ray diffraction analysis of the retained scale after the fourth cycle indicated the presence of Cr_2O_3 , spinel ($a_0 = 8.35 \text{ \AA}$), and possibly CoO . Microhardness tests indicated that the alloy was softer in the zone that was about 0.1 centimeter deep (fig. 4).

GE-1541

The iron-based alloy GE-1541 (14.54-wt % Cr, 4.14-wt % Al, 0.83-wt % Y, 0.0002-wt % C, and balance Fe) produced only a very small vapor deposit, which contained chromium and some iron. The deposit was too small for quantitative analysis. Additional oxidation for up to 96 hours did not increase the deposit. Apparently, the alumina scale that formed on the surface of the specimen protected it from further oxidation and reduced the loss of chromium by oxidation-volatilization.

Figure 7 shows the microstructure of GE-1541 in the as-received condition and after 96 hours of cyclic oxidation at 1200°C . The original grains were rather large and no growth could be observed in the oxidized specimen. However, during oxidation, a 0.02-centimeter-deep depletion zone formed that was void of YFe_9 precipitates. An X-ray diffraction analysis of the oxide scale on an oxidized specimen revealed the presence of $\alpha\text{-Al}_2\text{O}_3$, $Cr_2\text{O}_3$, and spinel. An attempt to perform an oxidation-volatilization experiment at 1300°C resulted in the melting and coalescence of YFe_9 along the grain boundaries of the matrix. The amount of vapor deposit obtained was still not sufficient for

analysis. The microhardness test did not reveal any drop in hardness of the alloy in the zone near the alloy - oxide-scale interface.

DISCUSSION

The volatilization of oxides and the determination of their vapor pressures have been the subjects of numerous investigations (refs. 5 to 12). Volatilization reactions have been proposed and formulas derived to express the vapor pressure of many oxides as a function of temperature. Also, methods were developed (refs. 13 to 15) for computing the oxide vapor pressures of simple systems as a function of oxygen pressure. Such computations require the knowledge of thermochemical data for all the elements, compounds, and vapor species that could exist during oxidation conditions.

In the oxidation of superalloys, only qualitative predictions could be made about the volatilization of oxides because of the complex nature of the problem. The results obtained in this investigation represent a quantitative determination of the content of metallic elements in oxide vapor deposits. They indicate which oxides volatilize preferentially from the oxide scales and thus permit us to establish which elements are lost from an alloy during high-temperature oxidation.

Calculating the ratio of the content in the vapor deposit (in percent) to the content in the original alloy (in percent) for the component metallic elements of an alloy gives certain numerical values that are a measure of such depletion. The following table is an attempt to rank the elements in the superalloy according to these numerical values, which are shown in parentheses. If for a given component element the number is greater than 1, its content in the alloy is being reduced. This ranking was made for the first and fourth oxidation cycles to show these values and to bring out the fact that they changed with time. The elements preferentially lost during oxidation were tungsten, molybdenum, manganese, niobium, and chromium. Chromium was the main element in all retained oxides except those on GE-1541. Tungsten, molybdenum, and niobium were detected in very small percentile amounts in the scales on alloys containing these elements. The preferential loss of these elements affected the hardness of the depleted zone in each of the alloys René 41, Stellite 6B, and Inconel 750. Figure 4 illustrates this phenomenon. The loss of the strengthening element occurred to a depth of 0.1 centimeter (0.04 in.). Porosity, the presence of which can be observed in figures 2(b), 3(b), 5(b), and 6(b), and the decrease in the amount of dispersed phase shown in figures 6(b) and 7(b) were also evidence of preferential loss of some component elements. Aluminum and silicon were never detected in the oxide vapor deposits from alloys containing these elements. The spallation of the oxide scales during cyclic oxidation of some superalloys might cause the composition of the oxide vapor to be different from that which could be obtained in the

Cycle	Ranking
Nichrome	
First	Mn(15. 89)>Cr(3. 30)>Ni(0. 09)
Fourth	Mn(7. 45)>Cr(4. 23)>Ni(0. 06)
Inconel 750	
First	Cr(5. 74)>Nb(4. 11)>Mn(3. 57)>Fe(0. 47) >Ni(0. 06)>Ti(0), Al(0), Si(0)
Fourth	Nb(5. 11)>Cr(4. 29)>Fe(1. 00)>Ni(0. 33) >Mn(0), Ti(0), Al(0), Si(0)
René 41	
First	Ti(5. 58)>Cr(3. 57)>Mo(1. 38)>Co(0. 05) >Ni(0. 01)>Al(0)
Fourth	Mo(9. 43)>Cr(0. 3)>Ti(0), Co(0), Ni(0), Al(0)
Stellite 6B	
First	W(5. 47)>Cr(1. 73)>Mo(1. 53)>Co(0. 35)>Fe(0)
Fourth	W(12. 08)>Mo(5. 73)>Cr(0. 69)>Co(0), Fe(0)

absence of spallation. However, these differences are not expected to be large enough to affect the general findings and conclusions.

SUMMARY OF RESULTS

The proportions of metallic components in the retained oxide scales of commercial Nichrome, Inconel 750, René 41, and Stellite 6B differ from those in the alloys as a result of selective oxidation and subsequent preferential volatilization of some component oxides. The compositions of their oxide vapor deposits differed from those of the oxide scales as a result of the preferential volatilization, which may in turn influence subsequent oxidation by removing some components from the scale. The elements preferentially lost during cyclic oxidation were tungsten, molybdenum, niobium, manganese, and chromium. This preferential volatilization caused the change in the hardness of the alloys, the formation of porosity, and the disappearance of precipitate phases in the depleted zone. Aluminum and silicon were never detected in the oxide vapor deposits of alloys containing these elements. Nickel, cobalt, and iron, the main component elements of superalloys, were found in the vapor deposits and in the corresponding oxide scales in percentile amounts considerably smaller than in the alloys. The tungsten, molybdenum, and niobium contents were much higher in the vapor deposits than in the alloys, but their

contents in the oxide scales were below those in the corresponding alloys. Chromium was the main component element in all the oxide scales except one (GE-1541). It was present in the form of Cr_2O_3 and spinels. Because the GE-1541 alloy formed a protective Al_2O_3 scale, it produced a small amount of oxide vapor deposit that was not sufficient for quantitative analysis.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, December 6, 1976,
505-01.

REFERENCES

1. Tedmon, C. S., Jr.: The Effect of Oxide Volatilization on the Oxidation Kinetics of Cr and Fe-Cr Alloys. *J. Electrochem. Soc.*, vol. 113, no. 8, Aug. 1966, pp. 766-768.
2. Kohl, Fred J.; and Stearns, Carl A.: Vaporization of Chromium Oxides from the Surface of TD-NiCr Under Oxidizing Conditions. *NASA TM X-52879*, 1970.
3. Kohl, Fred J.; and Stearns, Carl A.: Oxidation/Vaporization of Silicide Coated Columbium Base Alloys. *NASA TM X-67980*, 1971.
4. Leonard, Regis L.: SAVLOC, Computer Program for Automatic Control and Analysis of X-Ray Fluorescence Experiments. *NASA TN D-8398*, 1977.
5. Alcock, C. B.; and Peleg, M.: Vaporization Kinetics of Ceramic Oxides Around 2000°C . *Trans. J. Br. Ceram. Soc.*, vol. 66, 1967, pp. 217-232.
6. Graham, H. C.; and Davis, H. H.: Oxidation/Vaporization Kinetics of Cr_2O_3 . *J. Am. Ceram. Soc.*, vol. 54, no. 2, Feb. 1971, pp. 89-93.
7. Stearns, Carl A.; Kohl, Fred J.; and Fryburg, George C.: Oxidative Vaporization Kinetics of Cr_2O_3 in Oxygen from 1000° to 1300°C . *J. Electrochem. Soc.*, vol. 121, no. 7, July 1974, pp. 945-951.
8. Brewer, Leo; and Mastick, Donald F.: The Stability of Gaseous Diatomic Oxides. *J. Chem. Phys.*, vol. 19, no. 7, July 1951, pp. 834-843.
9. Grimley, R. T.; Burns, R. P.; and Inghram, Mark G.: Thermodynamics of the Vaporization of Nickel Oxide. *J. Chem. Phys.*, vol. 35, no. 2, Aug. 1961, pp. 551-554.

10. Israpilov, K. M. ; et al.: A Study of the Evaporation of Nickel and its Oxides in Vacuum and in an Atmosphere of Rarefied Oxygen by Mass-Spectroscopic Technique. Ukr. Phys., J., vol. 20, July 1975, pp. 1197-1199 (in Ukrainian).
11. Brewer, Leo: The Thermodynamic Properties of the Oxides and their Vaporization Process. Chem. Rev., vol. 52, no. 1, Feb. 1953, pp. 1-75.
12. Caplan, D. ; and Cohen, M.: The Volatilization of Chromium Oxide. J. Electro-chem. Soc., vol. 108, no. 5, May 1961, pp. 438-442.
13. Kellogg, H. H.: Vaporization Chemistry in Extractive Metallurgy. Trans. Am. Inst. Min. Eng., vol. 236, no. 5, May 1966, pp. 602-615.
14. Gulbransen, Earl A.: Thermochemistry and the Oxidation of Refractory Metals at High Temperature. Corros., vol. 26, no. 1, Jan. 1970, pp. 19-28.
15. Gulbransen, Earl A. ; and Jansson, Sven A.: Thermochemistry of Gas-Metal Reactions. Oxidation of Metals and Alloys, Am. Soc. Met., 1971, pp. 63-86.

TABLE I. - ANALYSIS OF VAPOR DEPOSITS OBTAINED DURING OXIDATION
OF COMMERCIAL NICHROME AT 1200° C IN AIR

Element	Composition of alloy	Composition of scale ^a after fourth oxidation cycle	Composition of vapor deposited during -			
			First oxidation cycle	Second oxidation cycle	Third oxidation cycle	Fourth oxidation cycle
Content, wt % of total metal						
Cr	19.26	b 67.3 ± 0.7	63.6 ± 3.6	64.3 ± 1.5	71.3 ± 1.1	81.4 ± 2.6
Mn	1.85	18.4 ± 0.2	29.4 ± 2.4	29.3 ± 0.9	22.3 ± 0.6	13.8 ± 1.5
Ni	78.89	14.3 ± 0.3	7.0 ± 1.4	6.4 ± 0.3	6.4 ± 0.3	4.8 ± 1.0

^aX-ray diffraction analysis of the retained oxide scale indicated the presence of the following phases: Cr_2O_3 (very strong), and spinel with $a_0 = 8.45 \text{ \AA}$ (medium).

^bThe \pm values are standard deviation (sigma).

TABLE II. - ANALYSIS OF VAPOR DEPOSITS OBTAINED DURING OXIDATION
OF INCONEL 750 AT 1200° C IN AIR

Element	Composition of alloy	Composition of scale ^a after fourth oxidation cycle	Composition of vapor deposited during -			
			First oxidation cycle	Second oxidation cycle	Third oxidation cycle	Fourth oxidation cycle
Content, wt % of total metal						
Ni	73.0	37.3 ± 0.2	4.4 ± 0.2	11.2 ± 1.1	20.1 ± 0.6	24.1 ± 0.2
Cr	15.0	46.2 ± 0.6	86.2 ± 1.4	78.4 ± 2.2	67.6 ± 1.6	64.5 ± 1.0
Fe	6.7	8.6 ± 0.2	3.2 ± 0.4	4.6 ± 1.1	6.0 ± 0.6	6.8 ± 0.4
Ti	2.4	6.8 ± 0.6	(b)	(b)	(b)	(b)
Nb	.8	(b)	3.7 ± 0.6	4.1 ± 0.7	4.6 ± 0.4	4.6 ± 0.2
Al	.8	(b)	(b)	(b)	(b)	(b)
Mn	.6	1.1 ± 0.2	2.5 ± 0.7	1.7 ± 1.1	1.7 ± 0.7	(c)
Si	.7	(b)	(b)	(b)	(b)	(b)
C	.04	-----	-----	-----	-----	-----

^aX-ray diffraction analysis of the retained oxide scale indicated the presence of the following phases: spinel with $a_0 = 8.40 \text{ \AA}$ (medium), Cr_2O_3 (very strong), and rutile structure (medium).

^bNot detected.

^cDetected but not determined.

TABLE III. - ANALYSIS OF VAPOR DEPOSITS OBTAINED DURING OXIDATION
OF RENÉ 41 AT 1200° C IN AIR

Element	Composition of alloy	Composition of scale ^a after fourth oxidation cycle	Composition of vapor deposited during -			
			First oxidation cycle	Second oxidation cycle	Third oxidation cycle	Fourth oxidation cycle
Content, wt % of total metal						
Cr	19	44.3 ± 0.7	67.8 ± 1.4	31.1 ± 0.9	23.7 ± 0.6	5.7 ± 0.7
Co	11	7.7 ± 0.3	.6 ± 0.15	(b)	.3 ± 0.05	(b)
Mo	10	3.4 ± 0.05	13.7 ± 0.3	64.7 ± 0.7	72.3 ± 0.6	94.3 ± 0.9
Ti	3.1	8.5 ± 0.6	17.2 ± 2.7	2.2 ± 0.5	1.7 ± 0.5	(b)
Al	1.5	(b)	(b)	(b)	(b)	(b)
Ni	55.3	36.1 ± 0.5	.7 ± 0.14	2.0 ± 0.07	2.0 ± 0.07	(b)
C	.09	-----	-----	-----	-----	-----

^aX-ray diffraction analysis of the retained oxide scale indicated the presence of the following phases: Cr_2O_3 (very strong), spinel with $a_0 = 8.30 \text{ \AA}$ (weak), and rutile structure (very weak).

^bNot detected.

TABLE IV. - ANALYSIS OF VAPOR DEPOSITS OBTAINED DURING OXIDATION
OF STELLITE 6B AT 1200° C IN AIR

Element	Composition of alloy	Composition of scale ^a after fourth oxidation cycle	Composition of vapor deposited during -			
			First oxidation cycle	Second oxidation cycle	Third oxidation cycle	Fourth oxidation cycle
Content, wt % of total metal						
Cr	30.0	57.0	51.9 ± 1.0	38.5 ± 1.0	20.8 ± 0.7	20.8 ± 0.7
W	4.5	4.0	24.6 ± 0.6	42.9 ± 0.7	64.1 ± 1.0	54.4 ± 0.6
Mo	1.5	.2	2.3 ± 0.4	4.5 ± 0.8	6.5 ± 0.3	8.6 ± 0.3
Fe	3.0	1.0	(b)	(b)	(b)	(b)
Co	59.8	37.8	21.2 ± 0.4	14.1 ± 0.3	8.6 ± 0.3	16.2 ± 0.4
C	1.2	-----	-----	-----	-----	-----

^aX-ray diffraction analysis of the retained oxide scale indicated the presence of the following phases: spinel with $a_0 = 8.35 \text{ \AA}$ (strong), Cr_2O_3 (very weak), and possibly CoO .

^bNot detected.

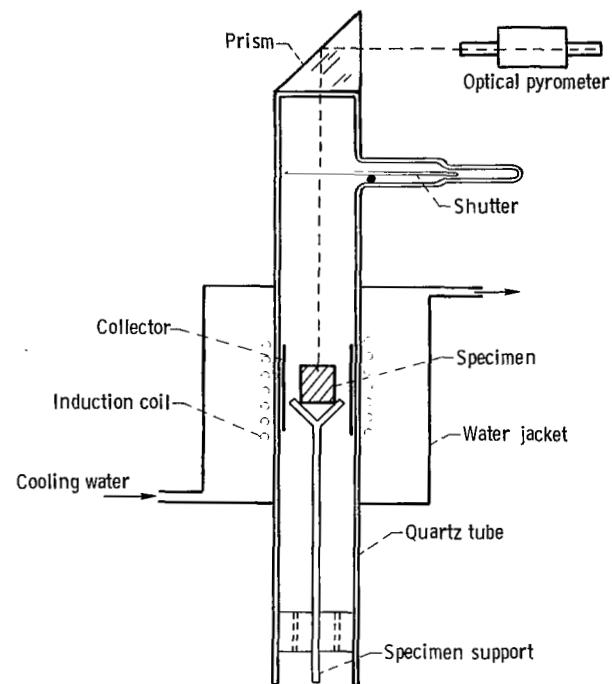
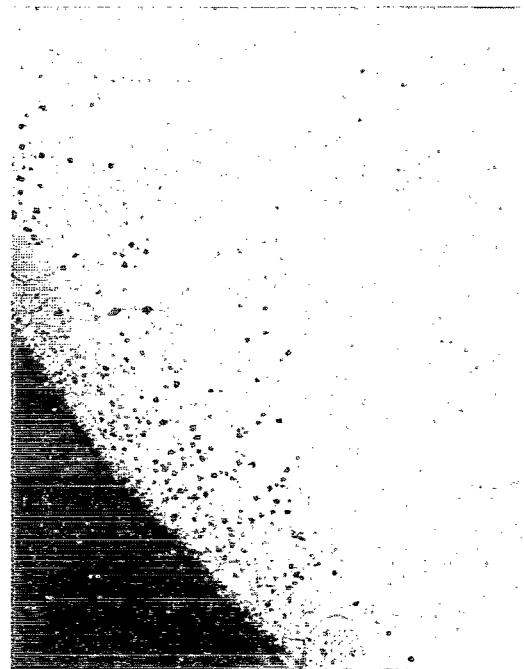


Figure 1. - Schematic illustration of oxidation-vaporization apparatus.



(a) As received.



(b) After oxidation.

Figure 2. - Microstructure of commercial Nichrome as received and after four 8-hour-long oxidation cycles at 1200°C. Etched; $\times 100$.



(a) As received.



(b) After oxidation.

Figure 3. - Microstructure of Inconel 750 as received and after four 8-hour-long oxidation cycles at 1200°C. Etched; $\times 100$.

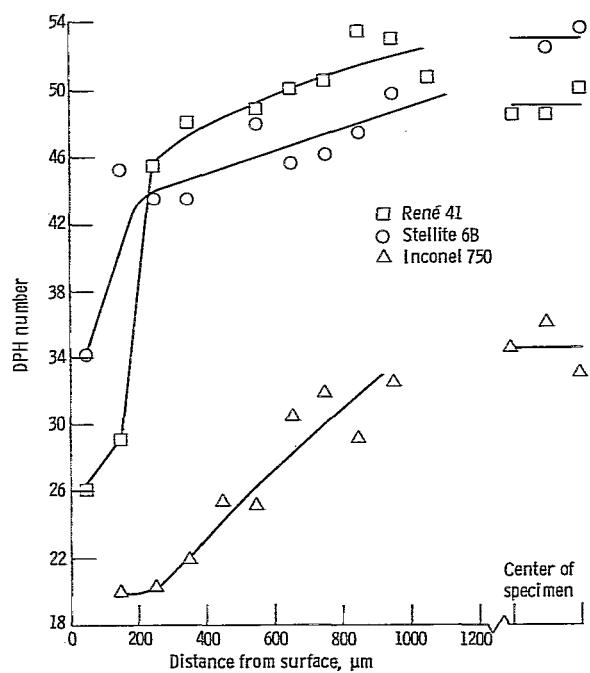
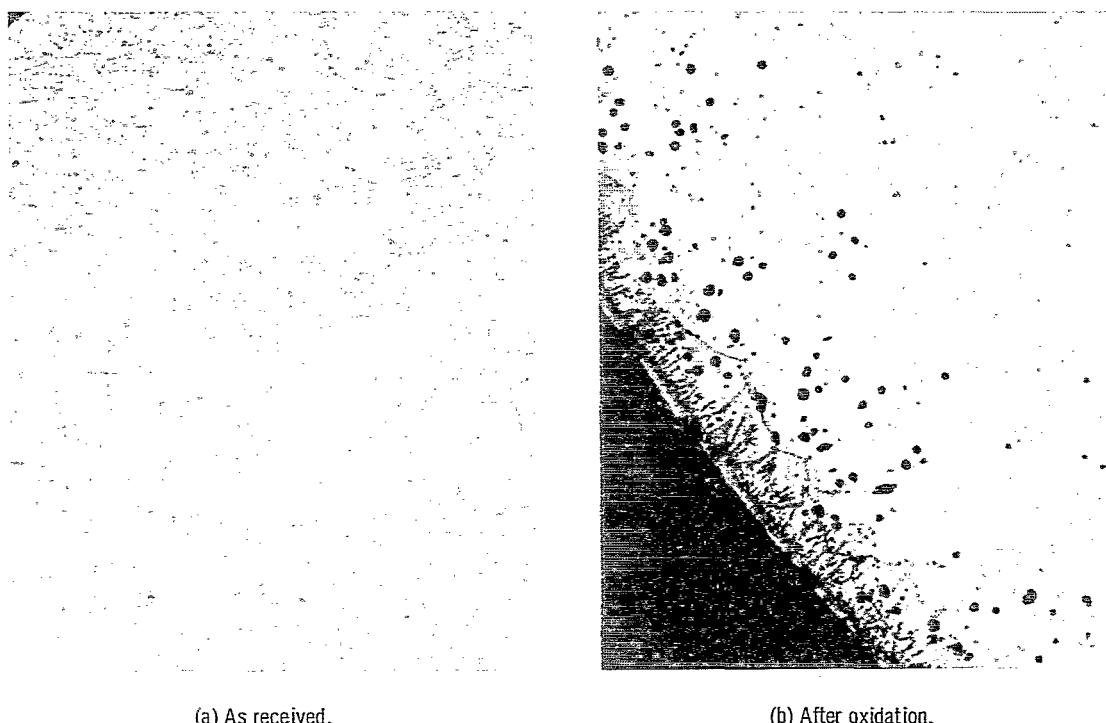


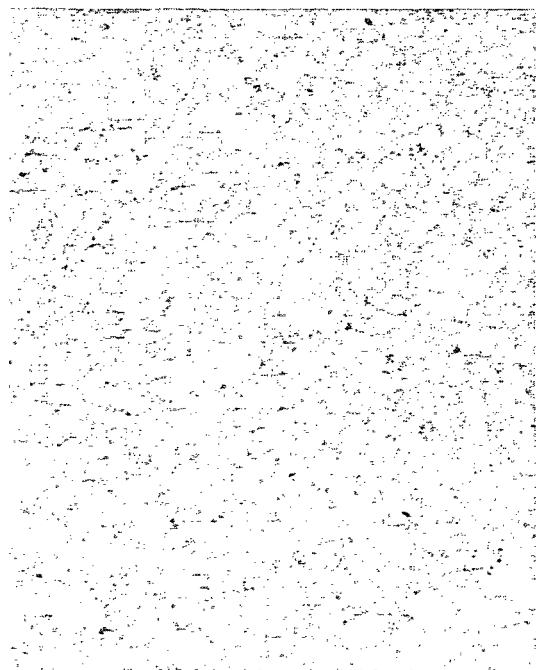
Figure 4. - Determination of hardness (DPH) in René 41, Stellite 6B, and Inconel 750 oxidized at 1200°C during four 8-hour-long cycles. Load, 200 grams.



(a) As received.

(b) After oxidation.

Figure 5. - Microstructure of René 41 as received and after four 8-hour-long oxidation cycles at 1200°C. Etched; $\times 100$.

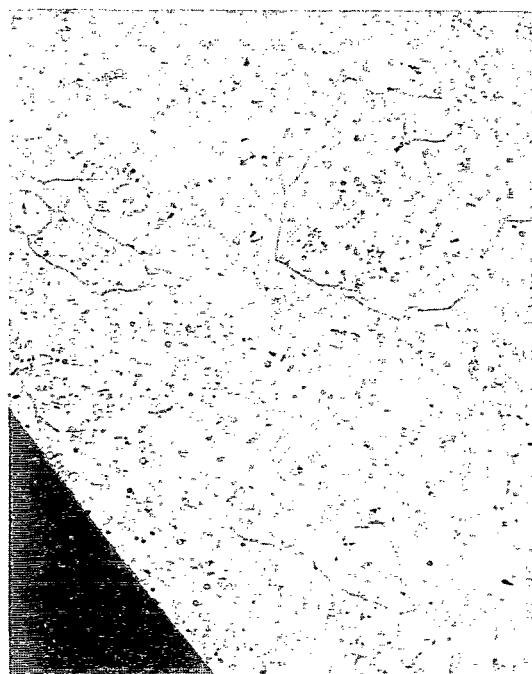


(a) As received.

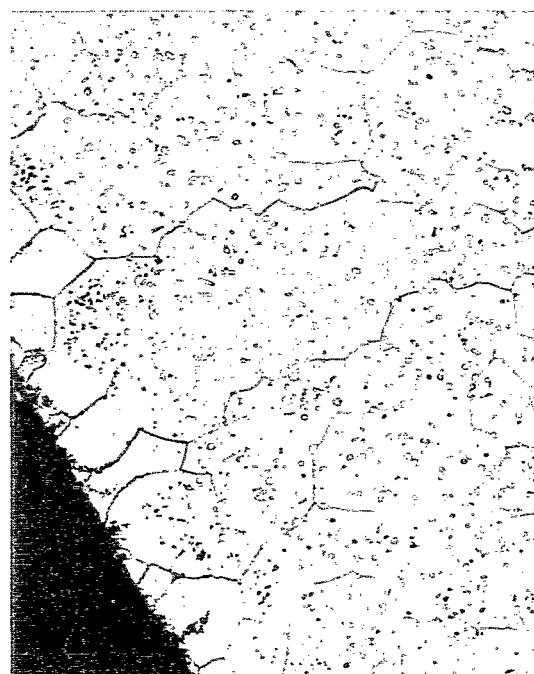


(b) After oxidation.

Figure 6. - Microstructure of Stellite 6B as received and after four 8-hour-long oxidation cycles at 1200°C. Etched; $\times 100$.



(a) As received.



(b) After oxidation.

Figure 7. - Microstructure of GE-154I as received and after twelve 8-hour-long oxidation cycles at 1200°C. Etched; $\times 100$.

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